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ATOMIC FORCE MICROSCOPY STUDIES OF MICROSTRUCTURE AND PROPERTIES OF SELF ASSEMBLED MONOLAYERS

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Self assembled monolayers are unique structures and have received considerable attention from microscopists seeking to image the predicted molecular level structure. More recently, practical engineering applications of SAMs have been proposed in areas ranging from corrosion barriers to adhesion promoters to lithographic resists.¹ While some of the applications of interest, most notably the lithographic resists, can be developed on substrates close to the ideal (such as single crystal Si wafers or thin epitaxial films); many others will require the coating of very non-ideal surfaces. These may range from materials in production lines such as Al or ferrous based metals to engineering polymeric materials. In this study we have taken a two-pronged approach to develop reliable systematic atomic force microscopy (AFM) techniques for the determination of both microstructure and properties of SAMs on various substrates of interest.

The majority of reported scanned probe microscopy studies on SAMs were performed on the n-alkanethiols (SH-(CH₂)_{n-1}-CH₃) on single crystal gold system and include both scanning tunneling microscopy (STM) and contact mode AFM on the molecular and atomic scales. There are several questions which need to be addressed based on previous studies. One is based on the importance of imaging force. It appears that the molecular structure of the SAM itself can be imaged at sufficiently low force, whereas at high forces, the gold lattice can be imaged through the SAM (or with the SAM pushed away by the tip).³ Thus, the tip sample interaction is clearly capable of modifying the observable structure during imaging and needs to be well characterized before extension of the technique to more complex or unknown structures.

This project is based on imaging the more complex and less studied SAM system of phosphonic acids on Al (with the native oxide). Three phosphonic acid compounds of varying alkyl chain lengths (short, medium, and long) are being used in this study; methylphosphonic acid (C1), dodecylphosphonic acid (C12), and octadecylphosphonic acid (C18), respectively. The Al surface is prepared by thermal evaporation onto glass slides in moderate vacuum. This system is more representative of commercially interesting SAMs than the thiol-Au system; but with much less known about it from previous investigations. In the phosphonic acid / Al system self-assembly of the monolayer occurs on a thin film of Al₂O₃, thus allowing for chemical bonding quite different from that of a noble metal such as gold. We have performed three studies on the phosphonic acid / Al system. The first studies the effect of imaging force on microstructure. A second explores the effect of alkyl chain length on microstructure. And a final study was performed to determine the effects of processing parameters (i.e. adsorption time) on the microstructure of phosphonic acids on aluminum.

High imaging forces, in both contact mode and tapping mode, altered the microstructure of the C12 monolayer. In contact mode AFM, the "root-like" structure ([figure 1](#), C12 monolayer) was not observed even at low contact forces. Further scanning of the same region using tapping mode AFM with low tapping force indicated that the "root-like" structure was displaced by the tip in contact mode and the microstructure was altered. Similarly, high tapping forces also resulted in altering the microstructure of the C12 monolayer.

The phosphonic acid / Al system was seen to exhibit an interesting, unexpected microstructure on the 10 to 100nm scale dependent on alkyl chain length and monolayer adsorption time. This microstructural dependence on alkyl chain length is shown in Figure 1. The microstructural dependence on adsorption time manifests itself primarily in the C1 and C12 monolayer as an increase in size and quantity of the "fiber-like" and "root-like" structures as the adsorption time was increased from 24 hours to 7 weeks. The microstructure of the C18 monolayer, however, showed little dependence on adsorption time.

These studies have allowed us to identify reliable AFM imaging modes and parameters for imaging the phosphoinic acid / Al system. When imaging under these conditions, AFM has proven to be viable technique for studying the microstructural dependence on alkyl chain length and processing parameters.

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Class	Functionality	Chemical Name
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Hydrophobic	C2	Ethyltriethoxysilane
Hydrophobic	C3	n-Propyltrichlorosilane
Hydrophobic	C3	n-Propyltrimethoxysilane
Hydrophobic	C4	n-Butyltrichlorosilane
Hydrophobic	C4	n-Butyltrimethoxysilane
Hydrophobic	C6	n-Hexyltrichlorosilane
Hydrophobic	C6	n-Hexyltrimethoxysilane
Hydrophobic	C8	n-Octyltrichlorosilane
Hydrophobic	C8	n-Octyltriethoxysilane
Hydrophobic	C10	n-Decyltrichlorosilane
Hydrophobic	C12	n-Dodecyltrichlorosilane
Hydrophobic	C12	n-Dodecyltriethoxysilane
Hydrophobic	C18	n-Octadecyltrichlorosilane

Hydrophobic	C18	n-Octadecyltriethoxysilane
Hydrophobic	C18	n-Octadecyltrimethoxysilane
Hydrophobic	C18	Glassclad-18
Hydrophobic	C20	n-Eicosyltrichlorosilane
Hydrophobic	C22	n-Docosyltrichlorosilane
Hydrophobic	Phenyl	Phenyltrichlorosilane
Hydrophobic	Phenyl	Phenyltriethoxysilane
Hydrophobic	Tridecafluorooctyl	(Tridecafluoro-1,1,2,2,-tetrahydrooctyl)-1-trichlorosilane
Hydrophobic	Tridecafluorooctyl	(Tridecafluoro-1,1,2,2,-tetrahydrooctyl)-1-triethoxysilane
Reactive	Acryl	3-Acryloxypropyltrichlorosilane
Reactive	Acryl	3-Acryloxypropyltrimethoxysilane
Reactive	Allyl	Allyltrichlorosilane
Reactive	Allyl	Allyltriethoxysilane
Reactive	Allyl	Allyltrimethoxysilane
Reactive	Bromo	3-Bromopropyltrichlorosilane
Reactive	Bromo	3-Bromopropyltrimethoxysilane
Reactive	Chlorobutyl	4-chlorobutyl dimethylchlorosilane
Reactive	(Chloromethyl)phenyl	(p-Chloromethyl)phenyltrichlorosilane
Reactive	(Chloromethyl)phenyl	(p-Chloromethyl)phenyltrimethoxysilane
Reactive	(Chloromethyl)phenyl	1-Trimethoxysilyl-2-(m,p-chloromethyl)phenylethane
Reactive	Chloromethyl	Chloromethyltrichlorosilane
Reactive	Chloromethyl	Chloromethyltriethoxysilane
Reactive	Chloroethyl	2-Chloroethyltriethoxysilane
Reactive	Chloropropyl	3-Chloropropyltrichlorosilane
Reactive	Chloropropyl	3-Chloropropyltrimethoxysilane
Reactive	Epoxy	3-Glycidoxypropyltrimethoxysilane
Reactive	Iodopropyl	3-Iodopropyltrimethoxysilane
Reactive	Isocyanato	3-Isocyanatopropyltriethoxysilane
Reactive	Mercapto	3-Mercaptopropyltrimethoxysilane
Reactive	Mercapto	3-Mercaptopropyltriethoxysilane
Reactive	Phosphino	2-(Diphenylphosphino)ethyltriethoxysilane
Reactive	Vinyl	Vinyltriacetoxysilane
Reactive	Vinyl	Vinyltrichlorosilane
Reactive	Vinyl	Vinyltriethoxysilane
Reactive	Vinyl	Vinyltrimethoxysilane
Polar/reactive	Amide	N-(Triethoxysilylpropyl)urea
Polar/reactive	Amino	N-(2-Aminoethyl)-3-aminopropyl trimethoxysilane
Polar/reactive	Amino	3-Aminopropyltriethoxysilane
Polar/reactive	Amino	3-Aminopropyltrimethoxysilane
Polar/reactive	Carbomethoxy	2-(Carbomethoxy)ethyltrichlorosilane
Polar/reactive	Carboxylic acid	N-[(3-Trimethoxysilyl)propyl]ethylenediamine triacetic acid trisodium salt
Polar/reactive	Cyano	3-Cyanopropyltrichlorosilane
Polar/reactive	Cyano	3-Cyanopropyltriethoxysilane
Polar/reactive	Chlorosulfonyl	2-(4-Chlorosulfonylphenyl)ethyltrichlorosilane
Polar/reactive	Chlorosulfonyl	2-(4-Chlorosulfonylphenyl)ethyltrimethoxysilane
Polar/reactive	Pyridyl	2-(Trimethoxysilyl)ethyl-2-pyridine

Polar/reactive	Pyrolle	N-(3-Trimethoxysilylpropyl)pyrolle
Polar/reactive	Quaternary Ammonium	N-Octadecyldimethyl-[(3-trimethoxysilyl)propyl]ammonium chloride
Polar/reactive	Ammonium	3-(N-Styrylmethyl-2-aminoethylamino)-propyl trimethoxysilane hydrochloride (40% in Methanol)
Polar/reactive	Quaternary Ammonium	N-Trimethoxysilylpropyl-N,N,N-trimethylammonium chloride

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